

Progress Report on U.S. Research on Materials and Test Methods

Takashi Kashiwagi

and

Vytenis Babrauskas

Building and Fire Research Laboratory
National Institute of Standards and Technology
Gaithersburg, MD 20899

I. MATERIALS

Two important recent trends which have started to affect the materials aspects of fires are non-halogenated flame retardant treatments and polymer recycling.

Brominated flame retardants have gained a major position in the worldwide plastics industry. To meet today's requirements in the electronic and electrical equipment industries, virtually every plastic requires flame retardancy. Due to negative publicity about dioxin and furan as possible degradation products, these retardants have received a negative public perception in Europe [1]. Some regulations on the use of certain types of these flame retardants will be introduced within the next several years. Although the use of halogenated flame retardants is still showing an upward trend, some concerns have been raised and there is a definite trend to seek alternatives for halogenated flame retardants. The possible introduction of future test methods on corrosivity of combustion products, as discussed in the Test Method Section, might further affect the use of halogenated flame retardants.

The huge waste volume of plastics is becoming a problem to modern societies. One of the solutions for the disposal of waste plastics is the use of landfills but the availability of landfill space is getting less. One alternative is the use of incinerators to burn the waste. Combustion products from halogenated plastics could ruin an incinerator by corrosion and they would become a public health problem without the use of a cleanup system for the incinerator exhaust. A more popular solution is the recycling of waste plastics. In Germany, even now, plastic products for packaging have to be taken back by the producer or retailer for recycling or disposal. This will be extended to include electronic scrap such as old computers, with an obligation to recycle as far as possible. Corresponding to these requirements, the labeling of all plastic components of computers to identify their material and manufacturers has already been implemented by IBM [2]. The emphasis on recycling might affect the selection of a base polymer and a flame retardant treatment on the basis of ease of recycling and also on durability. It could be possible that material selections might be more limited than in the past. Since the regulations as to fire safety are not eased by recycling, this could mean that the improvement in flammability properties for plastic materials might become more challenging.

One non-halogenated flame retardant approach is to form an intumescent char. It is well known that an intumescent char can act as a thermal barrier to protect polymer underneath. However, the optimum condition for achieving an effective intumescent char has not been well-understood;

Reprinted from: Proceedings of 12th Joint Panel Meeting of the UJNR Panel on Fire Research and Safety, Oct. 27 - Nov. 2, 1992, Produced by: Building Research Institute, Tsukuba, Ibaraki and Fire Research Institute, Mitaka, Tokyo, 1994.

there is a complex sequence of softening and melting of a polymer, degassing by blowing agents or the degradation of the polymer, followed by simultaneous intumescence and solidification of the polymer melt. A recent study has examined the effects of the metal oxide pigments, titanium dioxide and stannic oxide, with an intumescent additive system consisting of ammonium polyphosphate and a polyol type on the flammability of polypropylene [3]. A detailed study of a char formed was conducted by scanning electron microscopy, elemental composition analysis, and infrared spectroscopy. It was found that not only the amount of char formed but also the char structure are important to increase flame retardance. It is recommended that an effective flame retardant intumescent must have a cellular interior structure with a compact non-porous surface crust. The importance of the char structure on heat release rate was also found for a polycarbonate in studies at NIST. After the polycarbonate sample was burned at the external radiant flux of 40 kW/m^2 , about 40% of the initial weight was left behind as a residual char. This char was brittle and in the form of thin shells. Polycarbonate samples with silicone-containing compounds generated about the same amount of char at 40 kW/m^2 but a peak heat release rate was less than half of the polycarbonate sample without any treatment. The char from the silicone-containing polycarbonate sample consisted of many fine structures and also was spongy. At present the reasons why these char structures reduce peak heat release rate or increase limiting oxygen index is not clear. More detailed analysis of char and a heat transfer analysis through the char layer are needed to find an explanation.

Two new theoretical studies on the formation of char are progressing. One of them is the use of molecular dynamic simulations of the thermal degradation of polyethylene to identify factors which might be effective in reducing polymer flammability by promoting the formation of a residual char [4]. Computer movies of the calculated polymer chain trajectories during thermal degradation indicate that cross-linked polymers, such as those obtained from exposure of polyethylene to ionizing radiation will undergo further cross-linking when burned, eventually forming a high molecular weight, thermally stable char. More detailed information will be presented in this Session. The other theoretical study is the development of a model which can describe the transient behavior of the formation of intumescent char from a polymer which is heated by external radiation. This study is currently in progress at NIST by Howard Baum and Takashi Kashiwagi. The model is in the axisymmetric configuration and consists of the growth of bubbles from a blowing agent, followed by swelling of the polymer and a solidification reaction.

Numerous patents and studies on flame retardant additives have been published; the most recent review can be found in Ref.[5]. Only statistical data and recent results are summarized here. The U.S. consumption of flame retardant additives in thermoplastics and by thermoplastics type in 1990 [6] is shown in Fig.1 and 2, respectively. It shows that about 36% of flame retardant additives are non-halogenated additives. The types of flame retardant used in thermoplastics are shown in Fig.3 [6]. One interesting recent result is that the vapor phase flame retardant mechanism is not the whole story for antimony-halogen systems. A study of a brominated bisphenol A derivative with antimony oxide in nylon 66 showed that the main mode of action of the bromine additive with antimony oxide was in the condensed phase, via formation of antimony bromides which interacted with the nylon to form char [7].

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II. TEST METHODS

There is currently a huge number of available U.S. test methods for fire testing. For example, one such compilation of only the ASTM methods on fire [1] tabulates some 111 tests! Some of these methods are highly specialized to one particular industry, or are only useable for very approximate work. Others are obsolete, or nearly so. During recent years, great strides have been made, in fact, in developing useful, engineering-based methods. (Unfortunately, significant progress has not been made in eliminating the obsolete tests from the books; but their usage is, in most cases, diminishing or nearly non-existent.) Here, we will focus on the progress which has been made with the new engineering tools.

In this discussion, we will often be drawing close parallels between U.S. work and the work being done in other countries that are participating in ISO work. In many cases, we will show that U.S. efforts and mainstream ISO efforts have converged very closely. We will focus here solely on methods for *reaction-to-fire* testing; *fire resistance* test methods are not reviewed. The work will be reviewed according to the basic measurement categories.

Heat release rate

Over the last few years, heat release rate (HRR) has been recognized as the single most important variable describing the potential for fire hazard in most common scenarios [2]. This, not surprisingly, is therefore also the area in which the largest amount of research has taken place. Much of the new research is given in a recently published textbook on this subject [3]; this book also contains a very extensive bibliography, thus, only some highlights will be presented here.

(a) Large-scale fire tests

Building materials

ASTM first published in 1982 [4] a draft proposal for a standard room fire test. For various reasons, the method was not adopted as a standard. ISO (the International Organization for Standardization), however, has pursued the active standardization of a room fire test and just recently promulgated a standard method [5]. In the U.S., the most notable activity has been the organization of an international round robin, sponsored by Institute for Standards Research, which is an organization that has been established by ASTM for sponsoring certain types of research. The plan has been published [6] and much data have already been collected. Data from a number of bench-scale tests are also being collected in the program and it is expected to be completed in less than one year.

Furniture

Another development of much importance in the U.S. has been the re-writing and the widespread acceptance of the California T.B. 133 test for upholstered furniture. The room in which the testing is conducted was studied extensively by Parker and co-workers [7], leading to a conversion of the method to one that is HRR based instead of the temperature above the furniture. The revised standard is described by Nurbakhsh [8]. The newly-revised standard also permits testing to be done in an open calorimeter arrangement. ASTM are in the final stages of approving the T.B. 133 methodology as an ASTM standard. The National Fire Protection Association (NFPA), likewise, has been drafting a large-scale HRR method for furniture. The exact specifications of this are not yet clarified—it may be issued in a form permitting open calorimeter testing only, without giving the room/calorimeter choice that is in the California and in the ASTM standards. If this is the case, the arrangements are expected to be generally similar to the open calorimeter method used by Underwriters Laboratories (UL) [9].

Electric wire and cable

Length of char used to be the conventional variable for measuring wire and cable flammability. During the last few years it has increasingly become realized that HRR is a much more quantitatively useful variable. As a result, there has been an effort to introduce HRR measurements into wire and cable tests as performed at UL. The entire field of wire and cable reaction-

to-fire testing has recently been exhaustively reviewed by NIST [10]. Recent UL research is reported in [3].

(b) Bench scale tests

In the bench-scale HRR area, the Cone Calorimeter (ASTM E 1354 [11]) has been approved as ISO 5660 [12]. The world population of Cone Calorimeters stands at *ca.* 80. In Europe, there has been a strong focus on using the Cone Calorimeter to predict the behavior of building products. In the U.S., over the last few years the dominant efforts have been in the upholstered furniture and electric wire and cable areas. The furniture work has been reviewed in [3], while work on wire and cable is reviewed in [10]. To allow improved access to the large amount of research studies which have been carried out with the Cone Calorimeter, NIST have compiled a bibliography of all the citations known to us as of the end of 1991 [13].

Plastics development

One area where the use of the Cone Calorimeter has dramatically increased is in the development of improved plastics formulations. In the U.S., the primary performance tools used by the polymer chemist have been the limiting oxygen index test (LOI) and the UL 94 test. Neither of these tests can be considered an adequate engineering test for determining material fire properties. During the last year, we have seen a very significant change in the position of U.S. industry. In conferences and presentations where LOI and UL 94 results used to be presented nearly exclusively, performance data taken on the Cone Calorimeter are now routinely being given. This follows a similar trend which occurred in Europe a year or two earlier. On the issue of the non-applicability of the LOI test to assessment of fire performance, the first comprehensive review has been made [14].

Furniture

NFPA developed a Cone Calorimeter-based method (NFPA 264A) for measuring the HRR of upholstered furniture and mattresses several years ago. During this year, ASTM has also issued a method addressed to this same topic, ASTM E 1474. Because ASTM is the dominant organization in the U.S. issuing fire test standards, the availability of an ASTM method is expected to increase interest in bench-scale testing in this field.

Ignition

For general testing with a radiant exposure, ISO 5657 [15] has been the international method (no comparable method had ever been published in the U.S.) There is a strong movement, however, to replace the use of the ISO 5657 apparatus with the Cone Calorimeter, since it also provides this same needed measuring capability. Similarly, the LIFT apparatus for flame spread testing, discussed below, can also be used to obtain radiant ignition data. The ISO 5657 method provides for testing specimens which are in a horizontal orientation, the LIFT apparatus tests specimens oriented vertically, while the Cone Calorimeter is used for testing in either orientation.

With this in mind, there has been very little U.S. work addressed towards ignitability measurement alone. Conversely, for a majority of the Cone Calorimeter HRR studies that have been conducted, ignitability data are included.

Flame spread

The method for the study of against-the-wind flame spread that is used by NIST is the LIFT method, ASTM E 1321 [16]. The apparatus is identical with that used for measurement of surface flammability of bulkhead and deck finish materials specified by the International Maritime Organization, though the IMO method is different in the way the flame spread test is run and how the data is treated. The LIFT method is actively being pursued by ISO as a committee draft [17]. A round robin on this method is in progress. Data from this method are pertinent to a number of fire models including one developed by Cleary and Quintiere [18].

As an alternate to direct flame spread measurement methods are techniques to predict flame spread using data obtained in the Cone Calorimeter. Many researchers are using Cone Calorimeter data to predict with-the-wind flame spread rates. A model to predict against-the-wind flame spread has been put forth [3]. Especially attractive about such an approach is that both with-the-wind and against-the-wind spread rates may be encompassed by one test. A review of the current state of the art is given in [3].

Smoke

On a world-wide basis, there is still quite a bit of use of the obsolescent NBS single-chamber test (ASTM E 662) [19], while in Germany the dual-chamber method [20] finds some use. In the U.S., research on smoke in recent years has primarily been conducted in flow-through geometry tests. Thus, for bench-scale work the Cone Calorimeter is most commonly used, while for larger-scale studies open calorimeter arrangements are used.

Recently, Mulholland and associates examined the smoke production characteristics in the Cone Calorimeter when oxygen levels are lowered, going down all the way to the extinction point [21]. Studies have also been reported comparing smoke yields in bench-scale to values seen in larger-scale experiments [22][23]. Some extensive smoke data taken in bench scale experiments alone have also been reported [24][25]. An article describing some general engineering aspects of smoke measurement has also been published [26]. Smoke aspects of wire and cable flammability have been reviewed in [10].

Corrosive products

On a worldwide basis, the use of a corrosion test developed by the French telecommunications organization CNET has been the most prevalent. This method has been studied in the U.S. and has not been found to have enough minimum features to qualify as an adequate fire test [27]. A comprehensive study of the literature did not reveal the existence of any other methods better suited to the purpose [28]. Thus, in the U.S. most of the activity towards the development of a suitable test for corrosive products of combustion has centered around two new ASTM Task Groups which have been working rather independently in their efforts at test development. Task Group E5.21 T.G. 70 was formed first and has been studying the problem with a focus on building products. Task Group D09 T.G. 21 was formed more recently and has been focusing on electrotechnical equipment. The method of T.G. 70 [29][30][31] is based on a modification to the radiant toxicity apparatus, described in the next section below. The method of T.G. 21 [32][33] is based on a modification to the Cone Calorimeter. At the moment both methods are in the process of voting at ASTM and have not yet been approved. The method of T.G. 70 is intended to be proposed for international use for electrotechnical products, since the arrangements are clearly more satisfactory than in the prevalent CNET test.

It is important to note that, thus far, there have been no useful full-scale fire tests reported on corrosive products. Thus, the bench-scale tests being proposed are preliminary and must await validation in order to establish credibility for actual engineering work.

Toxic products

During the recent past, NIST has completed their development of a new engineering test method for measuring the toxic products of combustion. This apparatus was initially motivated by a radiant heat test method developed at the Weyerhaeuser Company [34] as a modification of the earlier cup furnace method developed at the National Bureau of Standards [35]. In 1987 the National Institute of Building Sciences (NIBS) commissioned Southwest Research Institute (SwRI) to develop this apparatus further [36]. Eventually, a NIBS test method was developed and put to vote at ASTM, which did not approve the method. NIST continued further development, both in its own laboratories and by funding SwRI.

The NIST method was published in 1991 [37] and has been placed on the agenda of both ASTM and NFPA. Earlier development work was published in conjunction with a set of validation data [38]. It is important to emphasize that one of the major reasons why credibility is placed in the new NIST-developed method is the fact that it has been successfully validated against real-scale data. Such successful validation is considered essential if a bench-scale engineering method is to be accepted as credible.

The final development of the NIST method was much facilitated by a cooperative arrangement between NIST and the Building Research Institute (BRI). Dr. S. Yusa was able to spend more than a year at NIST and contributed significantly towards making this new method feasible.

During the course of the development of the new radiant test apparatus, it was discovered that the yields of CO cannot be represented correctly in *any* bench-scale geometry yet examined. Thus, it became important to develop mathematical procedures for post-test correction of the numeric data, so that CO values associated with real-scale fires could be properly represented. Such a method was successfully developed [39].

Conclusions

The most significant new U.S. developments in the area of test methods have been in the area of toxicity and corrosivity of combustion products. Unlike in the other areas, here the methods available a few years ago were recognized as unsatisfactory. Thus, the development of new methods was anticipated. The development of the toxicity test method is considered complete and a satisfactory, validated method is available for use. The situation in corrosivity is much more preliminary. While two methods of significant interest have been reported during the last few years, none can be validated because real-scale corrosivity data do not exist. Furthermore, an engineering theory of corrosivity is not at hand either. This suggests that much further development will be needed until validated, quantitative engineering methods are viewed as being available in this field.

In the area of heat release, smoke, ignitability, and flame spread, competent, well-agreed-upon test methods already exist. Thus, during the last few years progress has consisted mainly of data gathering and also, in some cases, of new engineering theories for the use of such data.

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U.S. Consumption of Flame Retardant Additives in Thermoplastics, 1990

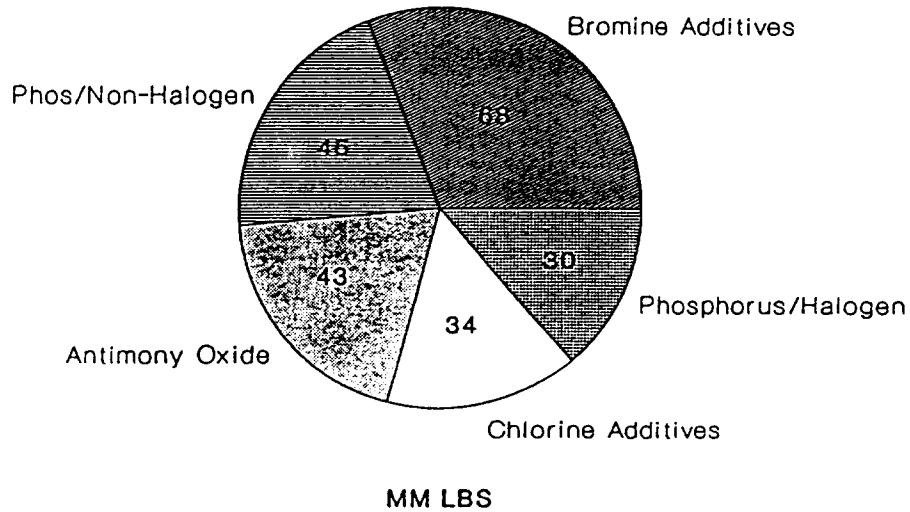


Figure 1

U.S. Consumption of Flame Retardant Additives by Thermoplastics Type, 1990

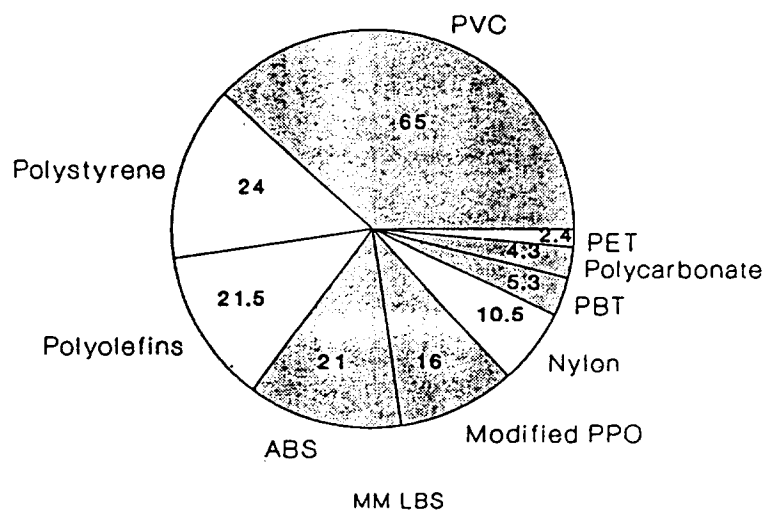


Figure 2

TYPE OF FLAME RETARDANT USED IN THERMOPLASTICS

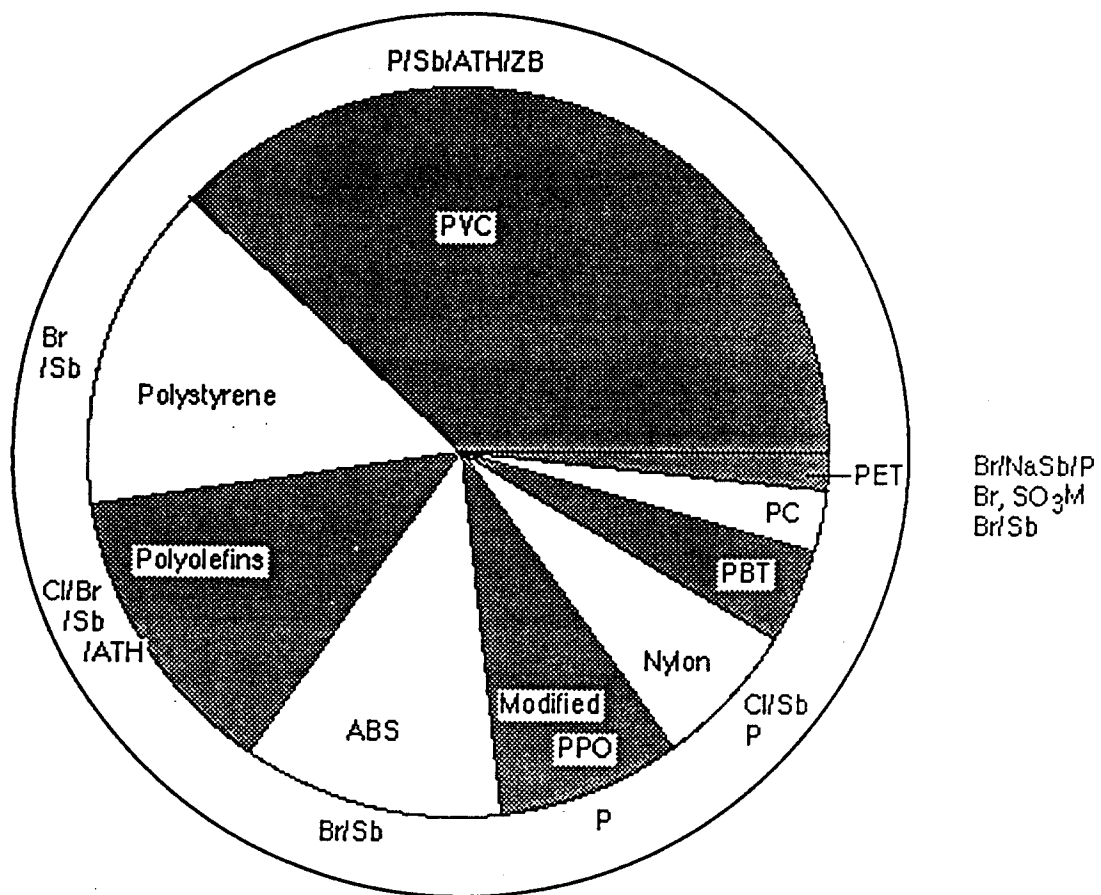


Figure 3